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EP 0867246 A1 EP 0773302 A1 WO 97/21509 A1
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(54) Abstract Title
Manufacturing castings from immiscible metallic liquids

(57) A method for forming a casting from a metallic alloy having at least two immiscible components, comprises the steps of

- heating the alloy to a temperature at least about its demixing temperature,
- transferring the alloy to a temperature-controlled twin-screw extruder 15, 16,
- cooling the alloy to a temperature at which said components become immiscible and
- operating the extruder to apply shear to the alloy in order to convert the alloy into a liquid suspension in which the minor liquid phase is dispersed in the major liquid phase,
- cooling the liquid suspension to the monotectic temperature or below and continuing to apply shear in order to form a semisolid slurry with a pre-determined volume fraction of the solid phase,
- transferring the semisolid slurry into a mould to form the casting. The casting formed comprises at least two immiscible components, the microstructure of the casting comprising a fine and uniform dispersion of the minor component in a matrix of the major component. A temperature-controlled extruder having a plurality of heating and cooling elements disposed along its long axis may alternatively be used in a modification. A further modification comprises apparatus for forming a casting from a metallic alloy having at least two immiscible components comprising a twin screw extruder capable of applying sufficient shear to said alloy when in a liquid state in its immiscibility gap in order to convert the alloy into a liquid suspension in which the minor liquid phase is dispersed in the major liquid phase, a shot assembly in fluid communication with the extruder, and a mould in fluid communication with the shot assembly.

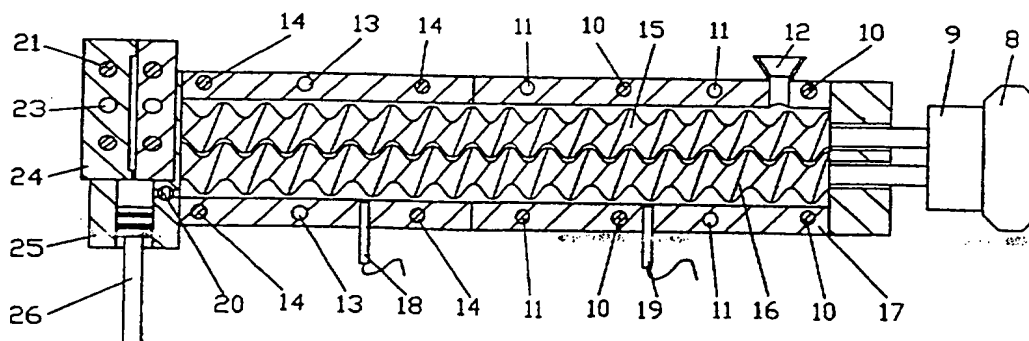
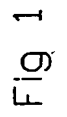
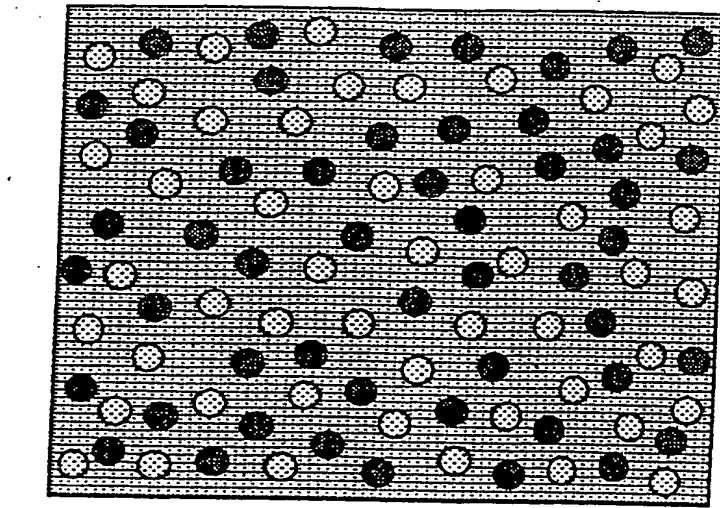


Fig. 3

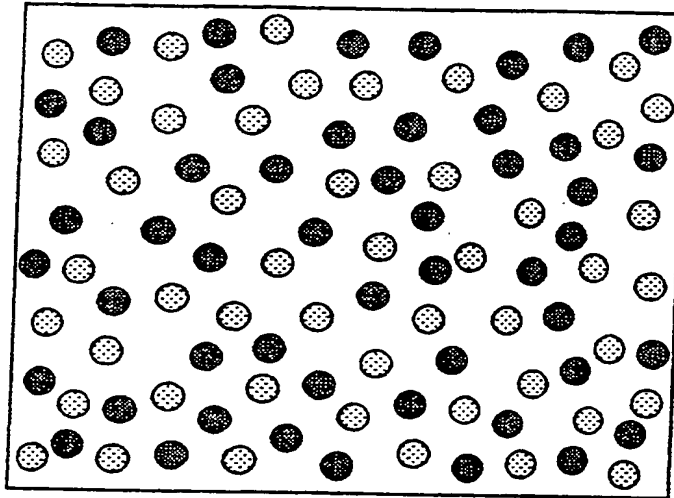
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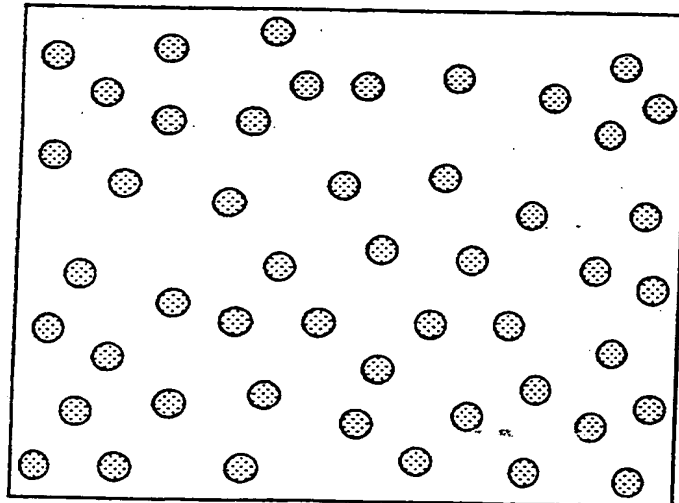
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(3) Monotectic solidification of L₁ and eutectic solidification of L₂.



(2) Formation of primary solid phase in L₁.
Further Stabilisation.



(1) Creation of a fine L₂ dispersion in L₁.
Initial Stabilisation.

Fig 2

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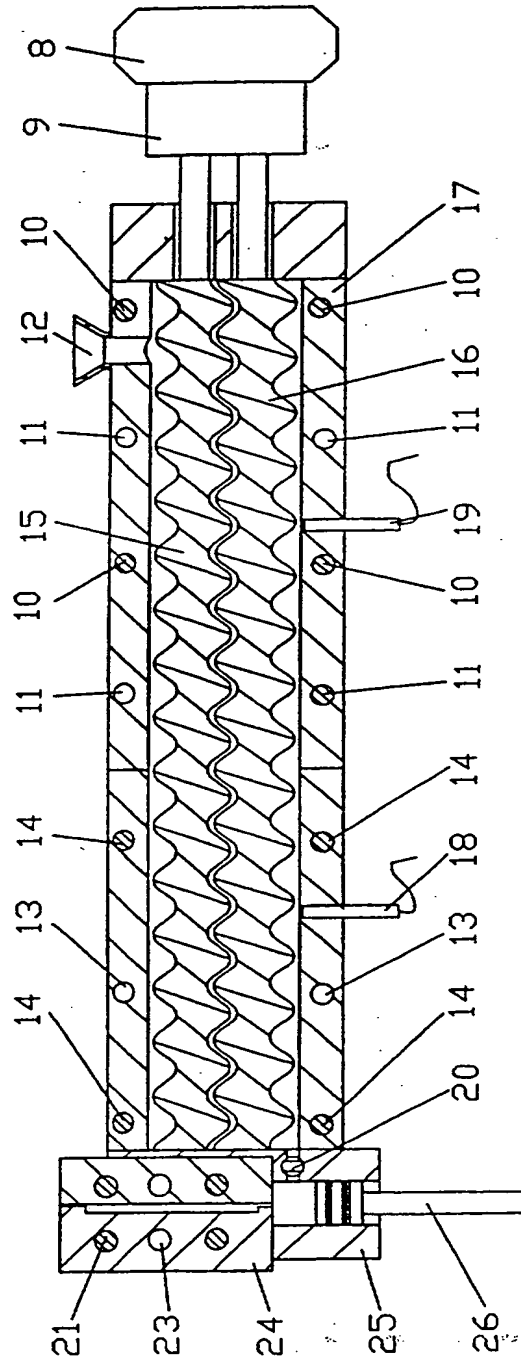


Fig. 3

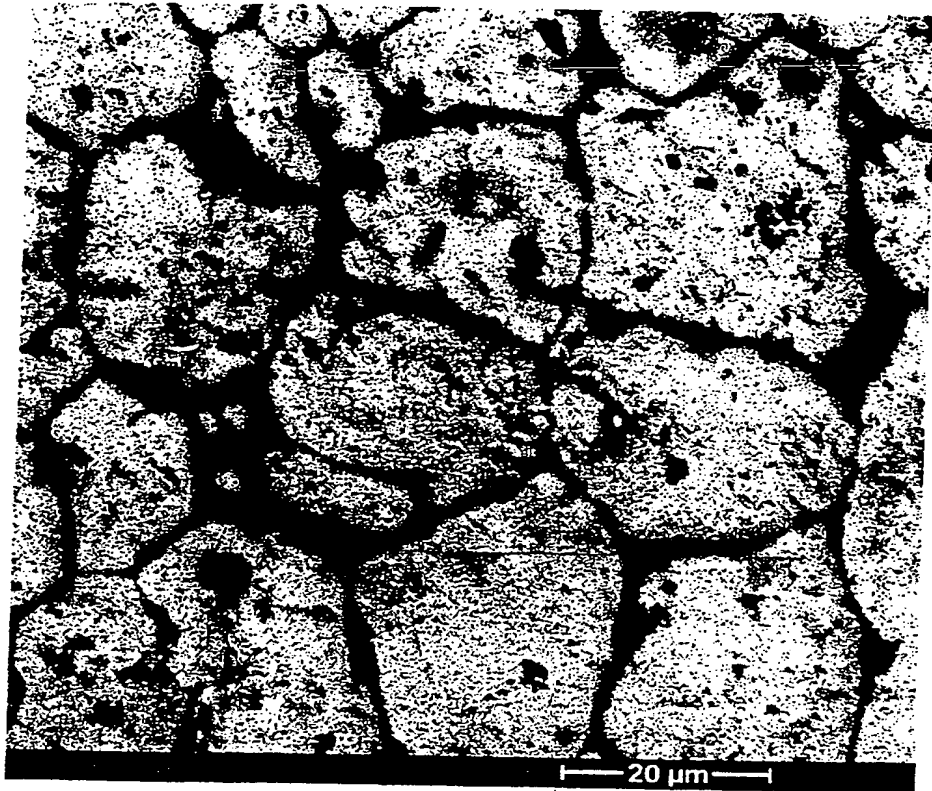


Fig 4

Process and Apparatus for Manufacturing Castings from Immiscible Metallic Liquids

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This invention relates to a process and apparatus for manufacturing castings from immiscible metallic alloys. At room temperature, one of the immiscible phases is finely and uniformly dispersed in the other immiscible phase throughout the casting section. The immiscible systems can be either binary or multi-component systems.

10

A large number of liquid alloys, such as Al-Pb, Al-In and Pb-Ga, exhibit a limited miscibility, i.e., the binary phase diagrams show a miscibility gap which represents the equilibrium between two liquids of different compositions. Those systems, often referred as the immiscibles, have great potential applications in advanced self-lubricating bearing systems, electrical contacts and superconducting devices.

15

Here, Al-Pb binary system is taken as an example to illustrate the microstructural requirement for bearing application. Usually, a material used for sliding bearings is of a composite nature with equiaxed inclusions of both hard and soft phases distributed in a mechanically strong matrix. The soft phase provides good embeddability, whereas the hard phase is responsible for the increased wear resistance. The tribological behaviour of such alloys is not only determined by the volume fraction of those phases but also by their particle size and distribution in the matrix. In the case of Al-Pb system for bearing application, it is desirable to have a microstructure in which the fine equiaxed soft Pb particles and a hard reinforcing phase (e.g., alumina) are uniformly distributed throughout the Al-alloy matrix.

20

25

Mixing the immiscible metallic systems has been a long-standing challenge to metallurgists and engineers. The efforts along this direction can go back as far as last

century. Unfortunately, it has not yet been possible technically to produce those alloys, because early in the cooling phase of the homogeneous liquid, those alloys separate quickly into two different liquids, one is Al-rich, the other is Pb-rich, owing to the large difference in density between Al and Pb. As described by Stoke's Law, the sedimentation velocity of the droplets increases as a function of the square of the droplet radius, therefore large droplets settle much more rapidly than the small ones. When droplets of different sizes and thus different sedimentation velocities collide, they coagulate to form even larger droplets which settle even more rapidly. Owing to this phenomenon, no casting process under terrestrial conditions has yet been able to produce the desired dispersion of the Pb particles in the Al matrix after solidification, even if extremely high solidification rates were achieved. These systems have gained renewed interests as a result of materials research in the outer space. Many tests have been performed during space experiments to achieve an appropriate phase distribution under microgravity conditions in the 1980s. The results, however, were rather disappointing, because even under microgravity conditions a coarse phase separation occurred. The origin of this coarse demixing was found to be Marogoni motion of the droplets, which is on the Earth superimposed on the gravity-driven sedimentation (Stoke's motion) and often hidden behind its action. However, having studied the results of space experiments, scientists started to utilise those unexpected findings in the early 1990s. A new strip casting process and a planar flow casting technique were developed recently in German Aerospace Research Establishment, DLR. In both processes, an artificial temperature gradient was created to introduce a controlled Marogoni motion, which was intend to counter-balance partially the gravity-driven Stoke's motion. Unfortunately, the experimental results were unsatisfactory, Pb was found to concentrate in the middle of the strip.

Therefore, it is a primary objective of this invention is to provide a process and apparatus for manufacturing castings from the immiscible metallic alloys, in which, at room temperature, one of the immiscible phases is finely and uniformly dispersed in the other immiscible phase throughout the casting section.

Another objective of the invention is to provide an apparatus and process which is specially adapted for mixing immiscible metallic alloys which are highly corrosive and erosive in their molten or semisolid state.

5

In a first aspect of the invention, there is provided a method for forming a casting from a metallic alloy having at least two immiscible components, comprising the steps of

- a) heating the alloy to at least about its demixing temperature,
- b) transferring the alloy to a temperature-controlled extruder which is capable of
- 10 operating at a sufficiently high shear to produce a suspension of the liquid alloy in which the minor liquid phase is dispersed in the major liquid phase and which is able to provide a positive pumping action,
- c) cooling the alloy to a temperature at which said components become immiscible and
- 15 d) operating the extruder to apply shear to the alloy in order to convert the alloy into a liquid suspension in which the minor liquid phase is dispersed in the major liquid phase,
- e) cooling the liquid suspension to the monotectic temperature or below and continuing to apply shear in order to form a semisolid slurry with a pre-determined
- 20 volume fraction of the solid phase,
- f) transferring the semisolid slurry into a mould to form the casting.

Preferably, the extruder is a twin screw extruder.

- 25 In a second aspect of the invention, there is provided a casting formed from a metal alloy comprising at least two immiscible components, wherein the microstructure of the casting comprises a fine and uniform dispersion of the minor component in a matrix of the major component.

In a third aspect of the invention, there is provided a temperature-controlled extruder, having a plurality of heating and cooling elements disposed along its long axis.

5 In a fourth aspect of the invention, there is provided apparatus for forming a casting from a metallic alloy having at least two immiscible components, comprising a twin screw extruder capable of applying sufficient shear to said alloy when in a liquid state in its immiscibility gap in order to convert the alloy into a liquid suspension in which the minor liquid phase is dispersed in the major liquid phase, a shot assembly in fluid communication with the extruder, and a mould in fluid communication with the shot
10 assembly.

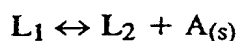
More particularly, the present invention relates to a method and apparatus for converting immiscible metallic liquid into a semisolid slurry and injecting subsequently the semisolid slurry into a die cavity for production of high integrity castings. The said method can
15 offer semisolid slurries with high enough viscosity to prevent coarse segregation in the immiscible system. The said apparatus and method can also offer net-shaped metallic castings with a fine and uniform dispersion of the minor phase in a matrix of the major phase. The said method comprises the steps of:

- 20 (a) feeding liquid metal at a temperature above T_X from a heated crucible into a twin-screw extruder through a feeder;
- (b) cooling the liquid metal rapidly into the miscibility gap in the first part of the extruder while being mechanically sheared by the twin-screw, converting the liquid alloy into a liquid suspension, wherein the minor liquid
25 phase is finely dispersed in the major liquid phase;
- (c) further cooling and shearing the liquid suspension to a temperature below T_M will allow the formation of a semisolid slurry with a pre-determined volume fraction of the solid phase;
- 30 (d) injecting the semisolid slurry at high velocity into a mould cavity for production of castings.

In order to facilitate the description of the present invention, a schematic binary phase diagram of an immiscible A-B system, as shown in Fig 1, is used to introduce the relevant terminology. The miscibility gap is represented by the curve MCF. For an alloy
 5 of a given composition, it is miscible above MCF and immiscible below MCF. The liquid separation occurs through the following reaction:



10 The maximum temperature on the miscibility gap is called the critical temperature, denoted as T_C . A monotectic reaction occurs at the monotectic temperature, T_M , to produce the solid phase $A_{(s)}$ from L_1 phase:



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and an eutectic reaction occurs at the eutectic temperature, T_E , to complete the solidification process:



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For a given alloy with X% of B, the liquid separation temperature will be T_X .

Briefly described, the above mentioned objectives are accomplished according to the present invention by providing an apparatus and process, rheomixer and rheomixing
 25 process, to produce castings from the immiscible liquid alloys. This is achieved by the following two-step strategy, as depicted in Fig 2:

(1) *Creation of a fine liquid dispersion by high shear mixing.* An artificial shear stress-strain field is applied continuously to the immiscible alloy during the cooling process
 30 from a temperature above T_X . This shear mixing action is so extensive that it can

override the demixing actions resulted from both Stoke's and Marogoni motions. Consequently, a fine homogeneous liquid dispersion is created at a temperature above T_M (Fig 2a).

- 5 (2) *Stabilisation of the fine liquid dispersion.* Although the fine liquid dispersion created by the high shear mixing action will slow down substantially the demixing process by both Stoke's and Marogoni motions due to the very much reduced droplet size, it is still unstable. With prolonged time, it will demix with increasing speed. However, the fine liquid dispersion can be further stabilised by shearing it at a temperature
10 below T_M to create a semisolid slurry (See Fig 1), the viscosity of which should be high enough that both Stoke's and Marogoni motions can no longer produce coarse separation (Fig 2b). In multi-component systems, the solid phase may be created at a temperature above T_M . In addition, the solid phase may be fine ceramic particles introduced externally to the alloy system at a temperature above T_M .

15

The invented rheomixer consists of a liquid metal feeder, a high shear twin-screw extruder, a shot assembly and a central control system. The rheomixing process starts from feeding liquid metal at a temperature above T_X from a heated crucible into a barrel through a feeder. The liquid metal is rapidly cooled into the miscibility gap in the first
20 part of the twin-screw extruder while being mechanically sheared the twin-screws, converting the liquid alloy into a liquid suspension, wherein the minor liquid phase is finely dispersed in the major liquid phase. Further cooling and shearing the liquid suspension to a temperature below T_M will allow the formation of a semisolid slurry with a pre-determined volume fraction of the solid phase dictated by accurate temperature
25 control. The semisolid slurry is then injected at a high velocity into a mould cavity by the shot assembly. The fully solidified casting is finally released from the mould. All these procedures are performed in a continuous cycle and controlled by a central control system.

Generally, the feeder is used to supply liquid alloy at the pre-set temperature to the twin screw extruder. The feeder can be a melting furnace or just a ladle.

Generally, the twin-screw extruder, consisting of a barrel, a pair of screws and a driving system, is adapted to receive molten alloy through an inlet located generally toward one end of the extruder. Once in the passageway of the twin-screw extruder, molten alloy is cooled rapidly to a predetermined processing temperature. The processing temperature can be either above or below T_M depending on the alloy systems. Also in the twin-screw extruder, the alloy is subjected to shearing. The shear rate is such that it is sufficient to create fine liquid droplets in a liquid suspension in the first stage of the cooling and to prevent the complete formation of dendritic shaped solid particles in a later stage of the cooling. The shearing action is inducted by a pair of co-rotating screws located within the barrel and is further invigorated by a helical screw flights formed on the body of the screws. Enhanced shearing is generated in the annular space between the barrel and the screw flights and between the flights of two screws. The positive displacement pumping action of the twin-screw can also cause the semisolid alloy to travel from the inlet of the extruder toward the outlet of the extruder, where it is discharged.

The interior environment of the twin-screw extruder is characterised by high wear, high temperature and complex stresses. The high wear is a result of the close fit between the barrel and the twin-screw as well as between the screws themselves. So a suitable material for the barrel and screws or any other components inside the extruder must exhibit good resistance to wear, high temperature creep and thermal fatigue. Meanwhile, the interior environment of the twin-screw extruder may also be highly corrosive and erosive if highly reactive alloys, such Al-alloys, are processed. After intensive tests and evaluation, the present invention has developed a novel machine construction which allows highly corrosive and erosive materials to be processed without any significant degradation of the machine itself.

The barrel of the twin-screw extruder is constructed with an outer layer of creep resistant first material which is lined with an inner layer of corrosion and erosion resistant second material. Preferably, the outer layer material is H11, H13, or H21 steels and the inner layer material is sialon. Bonding of the inner and outer layers is achieved by either
 5 shrunk fitting or with a buffer layer between the two. For small extruders, the barrel may be constructed from monolithic ceramics or tool steel with ceramic coatings, such as boron nitride.

The twin-screw is positioned within the passageway of the twin-screw extruder. The
 10 rotation of the twin-screw subjects the molten alloy to high shear and to translate the material through the barrel of the extruder. The screw is constructed with sialon components that are mechanically or physically bonded together to get its maximum resistance to creep, wear, thermal fatigue, corrosion and erosion. Additional components of the extruder, including the outlet pipe, outlet valve body and valve core, are also
 15 constructed from sialon. The twin-screw extruder is driven by either an electric motor or hydraulic motor through a gearbox to maintain the desired rotation speed. For small extruders, the twin-screws may be constructed from monolithic ceramics or tool steel with ceramic coatings, such as boron nitride.

20 The shot sleeve in the shot assembly of the rheomixer can receive the semisolid slurry from the extruder. The semisolid slurry in the shot sleeve can be injected at high speed to a die cavity by a fast moving piston through the cylinder.

Additional objectives and advantages of the invention will be set forth in the description
 25 which follows. The objectives and advantages of the invention may be realised and obtained by means of instrumentalities and combinations of particular points described in the appended claims.

A number of preferred embodiments of the invention are described in detail herein with
 30 reference to the drawings in which:

Fig 1 is a schematic binary phase diagram of the immiscible A-B system for introduction of the terminology used to describe the rheomixing process.

5 Fig 2 is a schematic illustration of the microstructural evolution during the rheomixing process. (1) initial stabilisation by creation of a fine L_2 dispersion in L_1 ; (2) further stabilisation by formation of a primary solid phase in L_1 ; (3) monotectic solidification of L_1 and eutectic solidification of L_2 .

10 Fig 3 is a schematic illustration of an embodiment of an apparatus for mixing the immiscible alloys and for producing castings according to the principles of the present invention.

Fig 4 shows the microstructure of the rheomixed Pb-20wt%Ga alloy. Ga phase is finely
15 and uniformly distributed in the Pb matrix.

In the description of the preferred embodiment which follows, a casting is produced by twin-screw rheomixer from a lead-gallium (Pb-Ga) binary system. However, the invention is not limited to Pb-Ga system and is equally applicable to any other types of
20 immiscible systems, such as Al-Pb, Al-In and Al-Bi. Furthermore, specific temperatures and temperature ranges cited in the description of the preferred embodiment are applicable only to Pb-Ga system, but could be readily modified in accordance with the principles of the invention by those skilled in the art in order to accommodate other alloy systems.

25

Fig 3 illustrates schematically a twin-screw rheomixer according to an embodiment of this invention. The rheomixing system has three sections: a liquid metal feeder, a twin-screw extruder and a shot assembly.

The feeder 12 is provided to receive liquid alloy with the predetermined temperature from an external source, such as a melting furnace or a ladle.

5 The twin-screw extruder has a plurality of heating elements 10, 14 and cooling elements 11, 13 dispersed along the length of the extruder. The matched heating elements 10, 14 and cooling elements 11, 13 form a series of heating and cooling zones respectively. The heating and cooling zones maintain the extruder at the desired temperature for processing immiscible alloys. For a rheomixing system designed for the Pb-Ga alloys, heating and cooling elements would maintain the extruder at a temperature around 280°C. The
10 heating and cooling zones also make it possible to maintain a complex temperature profile along the extruder axis, which may be necessary to achieve certain microstructural effects during rheomixing. The temperature control of each individual zone is achieved by balancing the heating and cooling power inputs by a central control system. The methods of heating can be resistance heating, induction heating or any other
15 means of heating. The cooling media may be water or gas depending on the process requirement. While only two heating/cooling zones are shown in Fig 3, the extruder can be equipped with between 1 to 10 separately controllable heating/cooling zones.

The extruder is also provided with twin-screw 15, 16 which are driven by an electric
20 motor or hydraulic motor 8 through a gear box 9. The twin-screw 15, 16 is designed to provide high shear rate which is necessary to achieve fine and uniform liquid suspension and fine and uniform solid particles. Different types of screw profiles may of course be used. In addition, any device which offers high shear mixing and positive displacement pumping actions may also be used to replace the twin-screw.

25

The semisolid slurry exits the extruder into a caster through a valve 20. The valve 20 operates in response to a signal from the central control system. The optional opening of valve 20 should match the process requirements.

Injection of the semisolid slurry is made by a piston 26 through the shot sleeve 25 into a mould cavity 24. The position and velocity of piston 26 are adjustable to suit the requirement by different processes, materials and final castings. Generally, the shot speed should be high enough to provide enough fluidity for complete mould filling, but not too high to cause air entrapment.

As shown in Fig 3, heating and cooling elements are also provided along the length of the shot sleeve 25 and the mould 24. In the preferred embodiment, the shot sleeve is preferably maintained at a temperature close to the extruder temperature to maintain the alloy in its predetermined semisolid state. The cooling rate of the solidifying alloy in the mould is controlled by the heating element 21 and cooling element 23.

In Fig 3, the barrel is made of tool steel with a sialon liner and the screws are monolithic sialon construction. However, the barrel and screws can be tool steels or any high temperature materials coated with any suitable ceramic materials, such as boron nitride.

Fig 4 illustrates the microstructure of the rheomixed Pb-20wt%Ga alloy. Ga phase is uniformly distributed in the Pb matrix. There was no coarse segregation observed in the casting.

The embodiment may also have a device attached to the extruder to supply protective gas in order to minimise oxidation. Such a gas may be argon or nitrogen.

Generally, the rheomixing system has a central control system to realise all the functions. Preferably, the control system is programmable so that the desired processing parameters may be achieved easily. The control system (not shown in Fig 3) may, for example, comprise a microprocessor which may be easily and quickly reprogrammed to change the processing parameters.

While this particular embodiment according to the present invention have been illustrated and described above, it will be clear that the invention can take a variety of forms and embodiments within the scope of the appended claims.

CLAIMS

1. A method for forming a casting from a metallic alloy having at least two immiscible components, comprising the steps of
 - 5 a) heating the alloy to a temperature at least about its demixing temperature,
 - b) transferring the alloy to a temperature-controlled twin-screw extruder,
 - c) cooling the alloy to a temperature at which said components become immiscible and
 - d) operating the extruder to apply shear to the alloy in order to convert the alloy into a liquid suspension in which the minor liquid phase is dispersed in the major liquid phase,
 - 10 e) cooling the liquid suspension to the monotectic temperature or below and continuing to apply shear in order to form a semisolid slurry with a pre-determined volume fraction of the solid phase,
 - 15 f) transferring the semisolid slurry into a mould to form the casting.
2. A method as claimed in claim 1, wherein, prior to being transferred into the mould, the slurry is transferred into a shot assembly which injects the slurry into the mould.
- 20 3. A method as claimed in claim 1 or 2, wherein a heated crucible is employed to transfer the alloy into the extruder.
4. A method as claimed in any preceding claim, wherein the alloy is sheared in step (e) at a sufficiently high rate to form semisolid slurry, the viscosity of which is high enough to prevent coarse segregation of the immiscible system.
- 25 5. A casting of a metal alloy comprising at least two immiscible components, wherein the microstructure of the casting comprises a fine and uniform dispersion of the minor component in a matrix of the major component.
- 30

6. A temperature-controlled extruder, having a plurality of heating and cooling elements disposed along its long axis.
- 5 7. An extruder as claimed in claim 6, wherein pairs of heating and cooling elements are matched to form heating and cooling zones.
8. An extruder as claimed in claim 6 or claim 7, which is a twin-screw extruder, or any equivalent device to supply high shear mixing and positive displacement
10 pumping action.
9. Apparatus for forming a casting from a metallic alloy having at least two immiscible components, comprising a twin screw extruder capable of applying sufficient shear to said alloy when in a liquid state in its immiscibility gap in
15 order to convert the alloy into a liquid suspension in which the minor liquid phase is dispersed in the major liquid phase, a shot assembly in fluid communication with the extruder, and a mould in fluid communication with the shot assembly.
10. Apparatus as claimed in Claim 9 wherein the extruder has a barrel and a pair of
20 screws, the inner surface of said barrel and the out surface of said screws are resistant to corrosion and erosion by liquid alloys, said twin screws each including a body having at least one vane thereon, said vane at least partially defining a helix around said body to propel the alloy through said barrel.
- 25 11. Apparatus as claimed in Claim 9 or 10 including drive means for rotating said twin-screw and shearing said immiscible metallic liquid at a rate sufficient to form semisolid slurry the viscosity of which is high enough to prevent the coarse segregation of the immiscible system, rotation of said twin screw by said drive means further causing said metal alloy to be transported from one end to another
30 end through said barrel.

12. Apparatus as claimed in any of claims 9 to 11, having temperature controllable means for transferring heat to said barrel, said twin screw and said metallic material therein such that said metallic material is in a semisolid state and at a temperature around its monotectic temperature T_M .
13. Apparatus as claimed in any of claims 9 to 12, having a control valve for discharging said metallic material from said extruder to a shot sleeve connected to a cylinder-piston assembly.
14. Apparatus as claimed in any of claims 9 to 13, wherein said inner surface of said barrel and the outer surface of said screw are coated with boron nitride or any other corrosion and erosion resistant materials.
15. Apparatus as claimed in any of claims 9 to 14, wherein the barrel and screws are made of monolithic sialon.
16. Apparatus as claimed in any of claims 9 to 15, further comprising a shot sleeve adapted to receive said metallic materials from said extruder, an piston for injecting said metallic materials in shot sleeve through a cylinder, a die cavity for receiving said metallic materials from said shot sleeve.
17. A method for forming a casting from a metallic alloy having at least two immiscible components substantially as hereinbefore described with reference to and as illustrated in the accompanying drawings.
18. Apparatus for forming a casting from a metallic alloy having at least two immiscible components substantially as hereinbefore described with reference to and as illustrated in the accompanying drawings.



Application No: GB 9922696.1
Claims searched: 1-5 & 17

Examiner: Pete Beddoe
Date of search: 17 December 1999

Patents Act 1977 Search Report under Section 17

Databases searched:

UK Patent Office collections, including GB, EP, WO & US patent specifications, in:

UK CI (Ed.Q): B3F (FCM, FCP, FCU)

Int CI (Ed.6): B22D (17/00, 17/30); C22C 1/00

Other: Online: WPI, EPODOC, JAPIO, CAS ONLINE

Documents considered to be relevant:

Category	Identity of document and relevant passage		Relevant to claims
X	EP 0867246 A1	(MAZDA) see esp col5 line 6 - col10 line 38 & fig 1a	1 & 5 at least
X	EP 0773302 A1	(HONDA) see esp embodiments & exs	5
X	WO 97/21509 A1	(THIXOMAT) see esp p4 line 29 - p11 line 15 & fig 1	1 & 5 at least
X	WO 95/34393 A1	(DOW) see esp p7 line 26- p11 line 33 & figs 3,4	1 & 5 at least
X	WO 90/09251 A1	(DOW) see esp p7 line 26 - p11 line 33 & figs 3,4	1 & 5 at least
X	US 5735333	(THE JAPAN STEEL) see esp col3 line 55 - col5 line 15 & figs	1 & 5 at least
X	US 5711366	(THIXOMAT) see esp cols 5-8 & figs 1,2	1 & 5 at least
X	US 5685357	(THE JAPAN STEEL) see esp col3 line 57 - col6 line 45, exs & figs 1,2	1 & 5 at least
X	US 5180450	(FERROUS) see esp col9 line 1 - col11 line 50	5

X	Document indicating lack of novelty or inventive step	A	Document indicating technological background and/or state of the art.
Y	Document indicating lack of inventive step if combined with one or more other documents of same category.	P	Document published on or after the declared priority date but before the filing date of this invention.
&	Member of the same patent family	E	Patent document published on or after, but with priority date earlier than, the filing date of this application.



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INVESTOR IN PEOPLE

Application No: GB 9922696.1
Claims searched: 1-5 & 17

Examiner: Pete Beddoe
Date of search: 17 December 1999

Category	Identity of document and relevant passage	Relevant to claims
X	US 4771818 (ALUMAX) see esp exs & claim 1	5
X	US 4694882 (DOW) see esp ex 1	1 & 5 at least
X	US 4694881 (DOW) see esp ex 1	1 & 5 at least
X	CA 2164759 A1 (INVENTRONICS) see esp claim 1	1 & 5 at least
X	High Temp. Mater. Processes, vol 14, pages 255-261, 1995, A Zhukov et al, " Properties of interface boundaries in segregating metallic mels", see abstract	5
X	Zh. Fiz. Khim., vol 49, pages 2570-2574, 1975, V I Kononenko et al, "Effect of liquid phase separation on the theromdynamic and kinetic properties of alloys", see abstract	5

X	Document indicating lack of novelty or inventive step	A	Document indicating technological background and/or state of the art.
Y	Document indicating lack of inventive step if combined with one or more other documents of same category.	P	Document published on or after the declared priority date but before the filing date of this invention.
&	Member of the same patent family	E	Patent document published on or after, but with priority date earlier than, the filing date of this application.

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